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# Indian Standard
# SPECIFICATION FOR NICKEL SALTS FOR ELECTROPLATING
## ( Second Revision )

UDC 661·874 : 621·357·7

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**INDIAN STANDARDS INSTITUTION**
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

*July* 1980

# *Indian Standard*

# SPECIFICATION FOR NICKEL SALTS FOR ELECTROPLATING

## *( Second Revision )*

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( *Continued on page* 2 )

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# AMENDMENT NO. 1 FEBRUARY 2005
# TO
# IS 1809 : 1979 SPECIFICATION FOR NICKEL SALTS FOR ELECTROPLATING

## *( Second Revision )*

( *Page* 10, *clause* A-4.4, *Formula* ) — Substitute '20.32' *for* '203.2'.

( CHD 1)

Reprography Unit, BIS, New Delhi, India

# *Indian Standard*

# SPECIFICATION FOR NICKEL SALTS FOR ELECTROPLATING

## *( Second Revision )*

## 0. FOREWORD

**0.1** This Indian Standard ( Second Revision ) was adopted by the Indian Standards Institution on 10 November 1979, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard was first published in 1961 and subsequently revised in 1969. However, with the progress made by the industry during the years, it was felt necessary to make the standard more comprehensive by including the requirements for nickel sulphamate. Thus the revision of this standard now covers the most commonly used nickel salts in the electroplating industry.

**0.3** In the formulation of this standard assistance has been derived from BS : 558 & 564-1970 ' Specification for nickel anodes, and nickel salts for electroplating ' issued by the British Standards Institution.

**0.5** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## 1. SCOPE

**1.1** This standard prescribes requirements and methods of sampling and test for the following nickel salts for electroplating:

a) Nickel sulphate,
b) Nickel chloride,
c) Nickel ammonium sulphate,

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*Rules for rounding off numerical values ( *revised* ).

d) Nickel carbonate, and

e) Nickel sulphamate.

NOTE — Nickel sulphamate is supplied in the form of a solution.

## 2. REQUIREMENTS

**2.1** Nickel salts and nickel sulphamate solution shall conform to the following description:

| NICKEL SALTS | GRADE | REQUIREMENTS | |
|---|---|---|---|
| Nickel sulphate | I and II | Emerald green crystals or green crystalline powder, free from extraneous matter | Mixture of hydrates, falling in the limits corresponding to the formula $NiSO_4.6H_2O$ and $NiSO_4.7H_2O$ |
| Nickel chloride | I and II | Yellowish-green crystals or powder, free from extraneous matter | Corresponding approximately to the formula $NiCl_2.6H_2O$. |
| Nickel ammonium sulphate | — | Blue-green crystals, free from extraneous matter | Corresponding approximately to the formula $NiSO_4.(NH_4)_2SO_4.6H_2O$. |
| Nickel carbonate | — | Light green powder, free from extraneous matter, soluble in acids | Corresponding approximately to the formula $NiCO_3.2Ni(OH)_2.4H_2O$. |

**2.1.1** Nickel sulphamate solution shall contain approximately 60 percent ( m/m ) of the salt $Ni(SO_3NH_2)_2.4H_2O$.

NOTE — The solution has a relative density of about 1·5.

**2.2** Nickel salts and nickel sulphamate solution shall also comply with the requirements given in Tables 1 and 2 respectively when tested in accordance with the methods prescribed in Appendix A. Reference to the the relevant clauses of Appendix A is given in col 9 and 4 of Tables 1 and 2 respectively.

## TABLE 1 REQUIREMENTS FOR NICKEL SALTS FOR ELECTROPLATING

( *Clause* 2.2 )

| SL No. | CHARACTERISTIC | NICKEL SULPHATE | | NICKEL CHLORIDE | | NICKEL AMMONIUM SULPHATE | NICKEL CARBONATE | METHOD OF TEST |
|---|---|---|---|---|---|---|---|---|
| | | Grade I | Grade II | Grade I | Grade II | | | |
| (1) | (2) | (3) | (4) | (5) | (6) | (7) | (8) | (9) |
| i) | *p*H of aqueous solution, not less than | 4·0 | 3·5 | 3·75 | 3·0 | 3·0 | — | A-2 |
| ii) | Insoluble matter, percent by mass, *Max* | 0·05 | 0·2 | 0·05 | 0·2 | 0·2 | 0·25 | A-3 |
| iii) | Nickel ( as Ni ), percent by mass, *Min* | 22·0 | 21·5 | 23·7 | 23·0 | 14·6 | 44·0 | A-4 |
| iv) | Cobalt ( as Co ), percent by mass, *Max* | 0·5 | 1 0 | 0·5 | 1·0 | 1·0 | 1·0 | A-5 |
| v) | Copper ( as Cu ), percent by mass, *Max* | 0·002 | 0 01 | 0·002 | 0·02 | 0·02 | 0·05 | A-6 |
| vi) | Heavy metals ( as Pb ), percent by mass, *Max* | 0·002 | 0·02 | 0·002 | 0·01 | 0·05 | 0·05 | A-7 |
| vii) | Iron ( as Fe ), percent by mass, *Max* | 0·005 | 0·02 | 0·01 | 0·05 | 0·05 | 0·10 | A-8 |
| viii) | Zinc ( as Zn ), percent by mass, *Max* | 0·005 | 0·01 | 0·005 | 0·01 | 0·01 | 0·05 | A-9 |
| ix) | Nitrates as ( $NO_3$ ), percent by mass, *Max* | 0·001 | 0·001 | 0·001 | 0·001 | — | 0·05 | A-10 |
| x) | Sulphate ( as $SO_4$ ), percent by mass, *Max* | — | — | — | — | — | 2·00 | A-11 |

## TABLE 2 REQUIREMENTS FOR NICKEL SULPHAMATE SOLUTION FOR ELECTROPLATING

( *Clause* 2.2 )

| Sl No.<br>(1) | Characteristic<br>(2) | Requirement<br>(3) | Method of Test<br>(4) |
|---|---|---|---|
| i) | *p*H of the aqueous solution as received, not less than | 4·0 | A-2 |
| ii) | Insoluble matter, g/l, *Max* | 1·0 | A-3 |
| iii) | Nickel ( as Ni ), percent ( *m/m* ), *Min* | 10·8 | A-4 |
| iv) | Coablt ( as Co ), percent ( *m/m* ), *Max* | 0·1 | A-5 |
| v) | Copper ( as Cu ), g/l, *Max* | 0·01 | A-6 |
| vi) | Heavy metals ( as Pb ), g/l, *Max* | 0·01 | A-7 |
| vii) | Iron ( as Fe ), g/l, *Max* | 0·01 | A-8 |
| viii) | Zinc ( as Zn ), g/l, *Max* | 0·01 | A-9 |
| ix) | Sulphate ( as $SO_4$ ), g/l, *Max* | 1·0 | A-11 |

Note — Subject to agreement between the purchaser and the supplier, concentration of 30 or 45 percent ( *m/m* ) may be accepted provided other requirements mentioned from [ Sl No. (v) to (ix)] remain same as above, except for nickel and cobalt content, which may reduce proportionately.

## 3. PACKING AND MARKING

**3.1 Packing** — The material shall be packed in air-tight containers lined with polyethylene liner as agreed to between the purchaser and the supplier.

**3.1.1** Nickel sulphamate solution shall be packed in containers made of a material like high density polyethylene.

**3.2 Marking** — The containers shall be legibly and indelibly marked with the following:

a) Name and grade, if any, of the material;
b) Net mass of the material ( in the case of nickel sulphamate solution, its volume, concentration and relative density shall be stated );
c) Name of the manufacturer or his recognized trade-mark, if any; and
d) Lot number and date of manufacture to enable the lot to be traced from the records.

**3.2.1** The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution ( Certification Marks ) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under, which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

## 4. SAMPLING

**4.1** The method of preparing representative samples of the material and the criteria for its conformity to this standard, shall be as prescribed in Appendix B.

## APPENDIX A

( *Clause* 2.2 )

## ANALYSIS OF NICKEL SALTS FOR ELECTROPLATING

### A-1. QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( *see* IS : 1070-1977* ) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

### A-2. DETERMINATION OF *p*H

**A-2.0 General** — Determination of *p*H shall be carried out at room temperature which shall be 27 ± 2°C.

**A-2.1 Apparatus**

**A-2.1.1** *p*H *Meter* — with glass electrode.

**A-2.2 Procedure**

**A-2.2.1** *Nickel Sulphate and Nickel Chloride* — Weigh accurately about 12·5 g of the material and dissolve in 125 ml of freshly boiled and cooled

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*Specification for water for general laboratory purposes ( *second revision* ).

distilled water and determine the *p*H value of the solution with the *p*H meter. Then rinse the electrodes with water and reserve the solution for further tests.

**A-2.2.2** *Nickel Ammonium Sulphate* — Weigh accurately about 12·5 g of the material and dissolve in 62·5 ml of freshly boiled and cooled distilled water and determine the *p*H value of the solution with the *p*H meter. Then rinse the electrodes with water and reserve the solution for further tests.

**A-2.2.3** *Nickel Sulphamate Solution* — Determine the *p*H value of the solution as received, with the *p*H meter.

## A-3. DETERMINATION OF INSOLUBLE MATTER

### A-3.1 Reagents

**A-3.1.1** *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

**A-3.1.2** *Dilute Sulphuric Acid* — 10 percent ( *v*/*v* ).

### A-3.2 Procedure

**A-3.2.1** *Nickel Sulphate* — Dilute the solution reserved in **A-2.2.1** to about 250 ml with water. Add one millilitre of dilute sulphuric acid, heat to boiling and filter through a tared sintered glass crucible ( G No. 4 ). Wash the residue thoroughly with hot water, dry at 105 ± 2°C to constant mass. Reserve the filtrate for further tests.

**A-3.2.2** *Nickel Chloride* — Dilute the solution reserved in **A-2.2.1** to about 250 ml with water. Add one millilitre of concentrated hydrochloric acid. Heat to boiling the proceed as in **A-3.2.1**. Reserve the filtrate for further tests.

**A-3.2.3** *Nickel Ammonium Sulphate* — Dilute the solution reserved in **A-2.2.2** to about 1 000 ml with water. Heat to boiling and proceed as in **A-3.2.1**. Reserve the filtrate for further tests.

**A-3.2.4** *Nickel Carbonate* — Weigh accurately about 12·5 g of the material and dissolve in 100 ml of dilute sulphuric acid. Heat to boiling and proceed as in **A-3.2.1**. Reserve the filtrate for further tests.

**A-3.2.5** *Nickel Sulphamate Solution* — Pipette 50 ml of the well mixed sample solution into a 600-ml beaker tared previously and weigh. Add 50 ml of water and proceed as in **A-3.2.1**. Reserve the filtrate for further tests.

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*Specification for hydrochloric acid ( *second revision* ).

### A-3.3 Calculation

a) Insoluble matter, percent by mass $= \frac{M_1}{M} \times 100$

b) Insoluble matter, g/l ( for nickel sulphamate solution ) $= M_1 \times 20$

where

$M_1$ = mass in g of the residue, and
$M$ = mass in g of the material taken for the test.

## A-4. DETERMINATION OF NICKEL

**A-4.0 General** — Nickel is determined by the gravimetric method using dimethylglyoxime.

### A-4.1 Reagents

**A-4.1.1** *Concentrated Sulphuric Acid* — *See* IS : 266-1977*.

**A-4.1.2** *Dilute Sulphuric Acid* — 10 percent ( *v/v* ).

**A-4.1.3** *Potassium Hydroxide Solution* — 10 percent ( *m/v* ).

**A-4.1.4** *Tartaric Acid*

**A-4.1.5** *Ammonium Hydroxide* — relative density 0·90.

**A-4.1.6** *Dimethylglyoxime Solution* — One percent ( *m/v* ) solution in rectified spirit.

### A-4.2 Preparation of Sample Solutions

**A-4.2.1** *Nickel Sulphate, Nickel Chloride and Nickel Carbonate* — Transfer the filtrates reserved in **A-3.2.1**, **A-3.2.2** and **A-3.2.4** respectively into 250-ml volumetric flasks, make up to the mark with water and mix.

**A-4.2.2** *Nickel Ammonium Sulphate* — To the filtrate reserved in **A-3.2.3** add 150 ml of potassium hydroxide solution and boil until ammonia is removed slowly, add 5 ml of concentrated sulphuric acid with stirring. Cool, transfer to a 250-ml volumetric flask, make up the volume with water and mix.

**A-4.2.3** *Nickel Sulphamate Solution* — Weigh accurately 10·0 g of the well-mixed sample solution, transfer to a 100-ml volumetric flask, make up to the mark with water and mix.

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*Specification for sulphuric acid ( *second revision* ).

### A-4.3 Procedure

**A-4.3.1** *Nickel Sulphate, Nickel Chloride, Nickel Ammonium Sulphate and Nickel Carbonate* — Pipette 20 ml of the sample solution ( 10 ml in the case of nickel carbonate solution prepared in **A-4.2**, into a 500-ml beaker, add 200 ml of water and 0·5 g of tartaric acid. Neutralize with ammonium hydroxide and make just acid with a few drops of dilute sulphuric acid. Heat to about 70°C and add 120 ml of dimethylglyoxime solution. Add ammonium hydroxide dropwise with stirring until ammonia smell persists. Then add 5 ml of ammonium hydroxide in excess with stirring and allow the precipitate to settle and cool to 25 to 30°C. Filter through a tared sintered glass crucible ( G No. 4 ) and wash the precipitate with water ( at about 25°C ) until free from chlorides and sulphates. Dry the precipitate at 105 ± 2°C to constant mass.

**A-4.3.2** *Nickel Sulphamate* — Pipette 30 ml of the solution prepared in **A-4.2.3** into a 400-ml beaker and dilute with 100 ml of water. Add, cautiously, 10 ml of concentrated sulphuric acid and heat to boiling for about half an hour. Cool, add 0·5 g of tartaric acid and proceed as in **A-4.3.1**.

### A-4.4 Calculation

$$\text{Nickel ( as Ni ), percent by mass} = \frac{203{\cdot}2\, M_1}{M}$$

where

$M_1$ = mass in g of the precipitate, and

$M$ = mass in g of the material or nickel sulphamate sample solution in the aliquot ( **A-4.3.2** ) taken for the test.

Note — Nickel content has been calculated on the basis of $NiSO_4.7H_2O$.

## A-5. DETERMINATION OF COBALT

**A-5.0 General** — Cobalt is determined by the iodometric method.

### A-5.1 Reagents

**A-5.1.1** *Potassium Hydroxide Solution* — 10 percent ( *m*/v ).

**A-5.1.2** *Glacial Acetic Acid*

**A-5.1.3** *Potassium Nitrite Solution* — 50 percent ( *m/v* ).

**A-5.1.4** *Ammonium Nitrate Solution* — 5 percent ( *m/v* ).

**A-5.1.5** *Standard Potassium Permanganate Solution* — approximately 0·1 N.

**A-5.1.6** *Dilute Sulphuric Acid* — 10 percent ( *v/v* ).

**A-5.1.7** *Potassium Iodide*

**A-5.1.8** *Standard Sodium Thiosulphate Solution* — approximately 0·1 N.

**A-5.1.9** *Starch Indicator Solution* — Triturate 5 g of pure starch and 0·01 g of mercuric iodide with 30 ml of water and slowly pour it with vigorous stirring into one litre of boiling water. Boil for 3 minutes, allow to cool and decant off the supernatant clear liquid.

### A-5.2 Procedure

**A-5.2.1** *Nickel Sulphate, Nickel Chloride, Nickel Ammonium Sulphate and Nickel Carbonate* — Pipette 20 ml of the sample solution, prepared in **A-4.2** into a 400-ml beaker, make alkaline with potassium hydroxide solution, add 2 ml of glacial acetic acid and heat to boiling. Add 10 ml of potassium nitrite solution. Stir, digest on a steam-bath for half an hour and allow to stand overnight. Filter through a tared sintered glass crucible ( G No. 4 ) and wash with small quantities of cold ammonium nitrate solution, containing 1 percent ( *v/v* ) glacial acetic acid, until free from nitrite. Place the crucible in the beaker in which precipitation was carried out, add 11 ml of potassium permanganate solution and 25 ml of dilute sulphuric acid and add sufficient water to cover the crucible. Heat to about 50°C until the yellow precipitate has dissolved, then cool and add 1 g of potassium iodide. Titrate the liberated iodine against standard sodium thiosulphate solution using starch as indicator. Carry out a blank titration with the same volume of potassium permanganate solution and dilute sulphuric acid and potassium iodide in the same manner as with the sample.

**A-5.2.2** *Nickel Sulphamate Solution* — Pipette 50 ml of the solution prepared in **A-4.2.3** into a 400-ml beaker and dilute with 100 ml of water. Add 10 ml of concentrated sulphuric acid, boil for about half an hour and allow to cool. Make alkaline with potassium hydroxide solution and proceed as in **A-5.2.1**.

### A-5.3 Calculation

$$\text{Cobalt ( as Co ), percent by mass} = \frac{0{\cdot}589\,4\, N\ (\, V_1 - V\,)}{M}$$

where

$N$ = normality of standard sodium thiosulphate solution,

$V_1$ = volume in ml of standard sodium thiosulphate solution used in the blank titration,

$V$ = volume in ml of the standard sodium thiosulphate solution used in the titration with the material, and

$M$ = mass in g of the nickel sulphamate sample solution or material in the aliquot taken for the test.

## A-6. DETERMINATION OF COPPER

**A-6.0 General** — Copper is determined by colorimetric method using potassium ferrocyanide.

### A-6.1 Apparatus

**A-6.1.1** *Nessler Cylinders* — 50-ml capacity ( *see* IS : 4161-1967* ).

### A-6.2 Reagents

**A-6.2.1** *Concentrated Hydrochloric Acid — See* IS : 265-1976†.

**A-6.2.2** *Mercuric Chloride Solution* — 1 percent ( *m/v* ).

**A-6.2.3** *Hydrogen Sulphide Gas*

**A-6.2.4** *Hydrogen Sulphide Water* — freshly prepared saturated aqueous solution.

**A-6.2.5** *Concentrated Nitric Acid — See* IS : 264-1976‡.

**A-6.2.6** *Potassium Ferrocyanide Solution* — 10 percent ( *m/v* ) in water.

**A-6.2.7** *Standard Copper Solution* — Prepared by dissolving 0·392 8 g of cupric sulphate ( $CuSO_4.5H_2O$ ) in water and making the solution to 1 000 ml. One millilitre of this solution is equivalent to 0·1 mg of copper ( as Cu ).

### A-6.3 Procedure

**A-6.3.1** *Nickel Sulphate, Nickel Chloride and Nickel Ammonium Sulphate* — Dissolve exactly 1·000 g of the material ( 5·000 g in the case of Grade I materials ) in sufficient water. Add 2 ml of concentrated hydrochloric acid and dilute with water to 50 ml. Add 2 ml of mercuric chloride solution and saturate with hydrogen sulphide. Filter through a small filter paper, wash with hydrogen sulphide water and ignite the precipitate in a porcelain crucible. Dissolve the residue by warming with 0·5 ml of concentrated nitric acid and a few drops of water and dilute to 10 ml filtering, if necessary. Dissolve 1 g of ammonium acetate in the solution and add 5 drops of potassium ferrocyanide solution. Carry out a control test, proceeding exactly as with the material, and using standard copper solution as follows:

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*Specification for Nessler cylinders.

†Specification for hydrochloric acid ( *second revision* ).

‡Specification for nitric acid ( *second revision* ).

a) For nickel sulphate, Grade I — 1 ml
b) For nickel sulphate, Grade II — 1 ml
c) For nickel chloride, Grade I — 1 ml
d) For nickel chloride, Grade II — 2 ml
e) For nickel ammonium sulphate — 2 ml.

**A-6.3.2** *Nickel Carbonate* — Weigh exactly 1·000 g of the material, transfer to 10 ml of water and heat gently adding concentrated hydrochloric acid till the material is just dissolved. Add 2 ml more of concentrated hydrochloric acid, dilute with water to 50 ml and proceed as in **A-6.3.1**, using 5 ml of standard copper solution in the control test.

**A-6.3.3** *Nickel Sulphamate Solution* — Transfer 5 ml of the well-mixed sample solution to 10 ml of water. Add 5 ml of concentrated sulphuric acid, evaporate to fumes and cool. Dilute with water to 50 ml and proceed as in **A-6.3.1** using 5 ml of standard copper solution in the control test.

**A-6.3.4** The requirements specified in Tables 1 and 2 shall be taken as having been satisfied if the intensity of colour produced with the material is not greater than that produced in the control test.

## A-7. DETERMINATION OF HEAVY METALS

**A-7.0 General** — Heavy metals are determined as lead by gravimetric method using ammonium molybdate as a precipitant. For Grade I of nickel salts, potassium chromate is used to see if any turbidity develops.

### A-7.1 Reagents for Grade I Nickel Salts

**A-7.1.1** *Sodium Acetate* — Solid.

**A-7.1.2** *Glacial Acetic Acid*

**A-7.1.3** *Potassium Chromate Solution* — Dissolve 1 g of potassium chromate in 100 ml of water.

### A-7.2 Reagents for Nickel Salts Other than Grade I

**A-7.2.1** *Ammonium Hydroxide* — 1 : 4 ( *v/v* ).

**A-7.2.2** *Dilute Sulphuric Acid* — 10 percent ( *v/v* ).

**A-7.2.3** *Hydrogen Sulphide Gas*

**A-7.2.4** *Hydrogen Sulphide Water* — Water, slightly acidified with sulphuric acid and saturated, with hydrogen sulphide gas.

**A-7.2.5** *Concentrated Nitric Acid* — *See* IS : 264-1976*.

**A-7.2.6** *Concentrated Sulphuric Acid* — *See* IS : 266-1977†.

**A-7.2.7** *Denatured Spirit* — *See* IS : 324-1959‡.

**A-7.2.8** *Ammonium Acetate Solution* — Dissolve 4 g in 1 litre of water acidified with 10 ml of acetic acid.

**A-7.2.9** *Ammonium Molybdate Solution* — Dissolve 4 g in 1 litre of water acidified with 10 ml of acetic acid.

**A-7.2.10** *Ammonium Nitrate Solution* — Dissolve 120 g in 1 litre of water containing 2 ml of ammonium hydroxide ( RD 0·9 ).

### A-7.3 Procedure

**A-7.3.1** *For Grade I Nickel Salts* — Dissolve 2·5 g of the material and 1 g of sodium acetate in 10 ml of water, add 5 drops of glacial acetic acid and 1 ml of potassium chromate.

**A-7.3.1.1** The requirements of Grade I of nickel salts as given in Table 1 shall be taken to have been satisfied if no turbidity is produced within 5 minutes.

**A-7.3.2** *For Nickel Salts Other than Grade I* — Neutralize 100 ml of the solution prepared in **A-4.2.1** and **A-4.2.2** with ammonium hydroxide and acidify with 5 ml of dilute sulphuric acid and pass hydrogen sulphide gas to saturation. Allow the precipitate to settle, filter through a pulp pad and wash the precipitate with hydrogen sulphide water. Transfer the filter pulp pad and precipitate to a 100-ml beaker, add 10 ml of concentrated nitric acid, 2 ml of concentrated sulphuric acid and evaporate to fuming and continue till organic matter is destroyed. Add more concentrated nitric acid if necessary. Cool, add 10 ml of water and fume again. Cool and dilute to 15 ml with water. Add 15 ml of denatured spirit and allow to stand till the lead sulphate precipitate has settled. Filter the precipitate and wash with denatured spirit. Dissolve the precipitate in 10 ml of boiling ammonium acetate solution and wash residue thoroughly with water. Heat the solution to boiling and add 10 ml of ammonium molybdate solution and boil for a few minutes to coagulate lead molybdate. Filter, preferably through a pulp pad, and wash

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*Specification for nitric acid ( *second revision* ).

†Specification for sulphuric acid ( *second revision* ).

‡Specification for ordinary denatured spirit ( *revised* ).

thoroughly with hot ammonium nitrate solution. Transfer to a weighed porcelain crucible and ignite at 600 to 650°C to constant mass.

**A-7-3.3** *For Nickel Sulphamate* — To the filtrate reserved in **A-3.2.5** add 5 ml of concentrated sulphuric acid, evaporate to fumes and cool. Add 10 ml of water, neutralize with ammonium hydroxide and proceed as in **A-7.3.2.**

### A-7.4 Calculation

a) Lead ( as Pb ), percent by mass $= \frac{0{\cdot}564\,4\,M_1}{M}$ , and

b) Lead ( as Pb ), g/l $= \frac{5{\cdot}64\,4\,M_1}{V}$

where

$M_1$ = mass in g of the precipitate, and
$M$ = mass in g of the material in the aliquot taken for the test, and
$V$ = volume in ml of the sample solution taken in **A-3.2.5.**

## A-8. DETERMINATION OF IRON

**A-8.0 General** — Iron is estimated by colorimetric method, using ammonium thiocyanate.

### A-8.1 Apparatus

**A-8.1.1** *Nessler Cylinders* — Two, of 50-ml capacity ( *see* IS : 4161-1967* ).

### A-8.2 Reagents

**A-8.2.1** *Concentrated Hydrochloric Acid* — *See* IS : 265-1976†.

**A-8.2.2** *Concentrated Nitric Acid* — *See* IS : 264-1976‡.

**A-8.2.3** *Ammonium Hydroxide* — 1 : 4 ( *v/v* ).

**A-8.2.4** *Dilute Ammonium Hydroxide* — 1 : 10 ( *v/v* ).

**A-8.2.5** *Dilute Hydrochloric Acid* — approximately 4 N.

**A-8.2.6** *Ammonium Persulphate*

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*Specification for Nessler cylinders.

†Specification for hydrochloric acid ( *second revision* ).

‡Specification for nitric acid ( *second revision* ).

**A-8.2.7** *Ammonium Thiocyanate Solution* — approximately 5 percent ( *m/v* ).

**A-8.2.8** *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [ $FeSO_4 . ( NH_4 )_2 SO_4 6H_2O$ ] in about 100 ml of water containing 10 ml of dilute sulphuric acid ( 10 percent, *v/v* ) and dilute to 1 000 ml. One millilitre of this solution contains 0·1 mg of iron ( as Fe ).

### A-8.3 Procedure

**A-8.3.1** *For Nickel Sulphate, Nickel Chloride, Nickel Ammonium Sulphate and Nickel Carbonate* — Dissolve exactly 1·000 g of the material ( 0·500 g in case of nickel carbonate ) in 20 ml of water ( adding just sufficient hydrochloric acid in case of nickel carbonate ). Add 2 ml of concentrated hydrochloric acid and 5 drops of concentrated nitric acid. Heat to boiling and add sufficient ammonium hydroxide to precipitate completely. Filter and wash the precipitate with dilute ammonium hydroxide until the washings are colourless. Dissolve any precipitate on the filter paper with 5 ml of hot dilute hydrochloric acid and dilute with water to about 20 ml. Transfer to a Nessler cylinder, add about 30 mg of ammonium persulphate and 3 ml of ammonium thiocyanate solution. Dilute to the mark and mix. Carry out a control test in the other Nessler cylinder proceeding as with the material, and using the following volumes of standard iron solution.

a) For nickel sulphate Grade I — 0·5 ml
b) For nickel sulphate Grade II — 2 ml
c) For nickel chloride Grade I — 1 ml
d) For nickel chloride Grade II — 5 ml
e) For nickel ammonium sulphate — 5 ml
f) For nickel carbonate — 5 ml

**A-8.3.2** *For Nickel Sulphamate* — Transfer 5 ml of the well mixed sample solution to 10 ml of water. Add 5 ml of concentrated hydrochloric acid, evaporate to fuming and cool. Add 1 ml of concentrated nitric acid, boil and proceed as in **A-8.3.1** using 5 ml of standard iron solution in the control test.

**A-8.3.3** The limits prescribed in Tables 1 and 2 shall, in respective cases, be taken as having not been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

## A-9. DETERMINATION OF ZINC

**A-9.0 General** — Zinc is estimated by colorimetric method using diphenylcarbazone.

### A-9.1 Apparatus

**A-9.1.1** *Nessler Cylinders* — Two, of 50 ml capacity ( *see* **A 8.1.1** ).

**A 9.1.2** *Ammonium Thiocyanate Solution* — 10 percent ( *m/v* ), filtered.

**A-9.1.3** *Ammonium Thiocyanate Wash Solution* — 1 percent ( *m/v* ). Just before use, add 5 ml of acridine hydrochloride solution to 200 ml of the wash solution.

**A-9.1.4** *Dilute Hydrochloric Acid* — 50 percent ( *v/v* ).

**A-9.1.5** *Ammonium Chloride Solution* — 20 percent ( *m/v* ).

**A-9.1.6** *Sodium Carbonate Solution* — approximately 1 N.

**A-9.1.7** *Hydrogen Peroxide* — Strength, 20 percent ( *m/m* ).

**A-9.1.8** *Ammonium Chloride Wash Solution* — 1 percent ( *m/v* ), containing a few drops of sodium carbonate solution.

**A-9.1.9** *Potassium Chloride Wash Solution* — 0·25 percent ( *m/v* ).

**A-9.1.10** *Acetone* — *See* IS : 170-1976*.

**A-9.1.11** *Diphenylcarbazone Solution* — Dissolve 2 g in one litre of pure amyl alcohol.

**A-9.1.12** *Amyl Alcohol* — of high purity. ( To test the purity, add 1 ml of diphenylcarbazone solution to 9 ml of amyl alcohol. The resulting solution should be pale yellow. If there is any trace of pink colour, redistill the amyl alcohol. )

**A-9.1.13** *Cobalt Sulphuric Solution* — 1 percent ( *m/v* ).

**A-9.1.14** *Standard Zinc Solution* — Dissolve 0·879 6 g of zinc sulphate heptahydrate ( $ZnSO_4 7H_2O$ ) in water, add 10 ml of dilute sulphuric acid ( approximately 4 N ) and dilute to 1 000 ml. Dilute 50 ml of this solution to 1 000 ml. One millilitre of the diluted solution contains 0·01 mg of zinc ( as Zn ).

### A-9.2 Procedure

**A-9.2.1** *For Nickel Sulphate, Nickel Chloride, Nickel Ammonium Sulphate and Nickel Carbonate* — Dissolve exactly 1·000 g of the crushed and well-mixed material ( 0 200 0 g in case of nickel carbonate ) in 5 ml of water ( adding just sufficient dilute hydrochloric acid in case of nickel carbonate ). Add 5 ml of cobalt sulphate solution, followed by 25 ml of

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*Specification for acetone ( *second revision* ).

acridine hydrochloric solution and mix. Add slowly, with constant stirring 25 ml of ammonium thiocyanate solution. Shake vigorously to coagulate the green cobalt complex. Allow to stand for 10 minutes and filter on a hardened filter paper ( Whatman No. 541 is suitable ). Wash the beaker 3 times and the paper 4 times with ammonium thiocyanate wash solution. Place the beaker in which precipitation was carried out under the funnel. Pierce a hole in the filter paper and wash the precipitate with hot water. Pour 10 ml of ammonium chloride solution through the funnel and wash several times with hot water until the paper is colourless. Keep the volume of water used for washing as low as possible.

**A-9.2.1.1** Warm the beaker containing the precipitate on a hotplate, shaking frequently until a clear solution is obtained. To the warm solution ( 60° to 70°C ), add from a pipette 10 ml of sodium carbonate solution and then 5 ml of hydrogen peroxide. Allow to stand warm for 5 minutes and then allow to cool for 20 minutes ( *see* NOTE ). Filter off the acridine and ferric hydroxide on a hardened filter paper ( Whatman No. 541 is suitable ) and collect the filtrate in a beaker marked at 100 ml. Wash several times with cold ammonium chloride wash solution. Dilute the filtrate to 100 ml. Add 5 ml of potassium cyanide solution. Shake and add 5 ml of acetone. Transfer the solution to a 250-ml separating funnel. Add 20 ml of diphenylcarbazone solution and extract for about 30 seconds. Run off the aqueous layer and transfer the other layer into a dry beaker. Extract the aqueous layer with a further 20 ml portion of diphenylcarbazone solution and combine the two extracts. Pipette 2 ml from the 40 ml of the extract in a Nessler cylinder and dilute to 10 ml with amyl alcohol. Mix thoroughly. Carry out a control test in the same manner using 10 ml ( 5 ml in case of Grade I material ) of standard zinc solution in place of the material.

NOTE — It is essential that the time of standing with hydrogen peroxide is strictly adhered to if cobalt interference is to be avoided.

**A-9.2.2** *For Nickel Sulphamate* — Dilute 5 ml of the well mixed sample solution to 100 ml with water in a volumetric flask. To 20 ml of this add 2 ml of concentrated hydrochloric acid and evaporate to dryness. Redissolve in 10 ml of dilute hydrochloric acid, add 5 ml of cobalt sulphate and proceed as in **A-9.3.1** and **A-9.3.1.1**.

## A-10. DETERMINATION OF NITRATES

### A-10.1 Reagents

**A-10.1.1** *Sodium Carbonate* — *See* IS : 296-1974*.

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*Specification for sodium carbonate, anhydrous ( *second revision* ).

**A-10.1.2** *Concentrated Sulphuric Acid* — *See* IS : 266-1976*.

**A-10.1.3** *Dilute Sulphuric Acid* — 20 percent ( *v/v* ).

**A-10.1.4** *Indigo Carmine Solution* — Dissolve 0·10 g of indigo carmine, previously dried for 2 hours at 105 ± 2°C, in a mixture of 10 ml of concentrated sulphuric acid in 80 ml of water and dilute to 100 ml.

**A-10.2 Procedure**

**A-10.2.1** *Nickel Sulphate and Nickel Chloride* — Weigh accurately 500 g of the material and dissolve in 50 ml of water. Add sodium carbonate with constant stirring till precipitation is complete. Add a pinch in excess. Evaporate to dryness and finally ignite gently avoiding any spurting. Cool, take up the residue with 25 ml of water and filter. To 5 ml of the filtrate add concentrated sulphuric acid dropwise till effervescence ceases, then add 0·1 ml of indigo carmine solution and 5 ml of concentrated sulphuric acid.

**A-10.2.2** *Nickel Carbonate* — Weigh accurately 0 10 g of the material and dissolve in minimum quantity of dilute sulphuric acid. Add sodium carbonate with stirring till precipitation is complete. Add a pinch in excess and proceed as in **A-10.2.1**.

**A.10.2.3** The material shall be taken as having satisfied the requirements of the test if the blue colour does not stay longer than 30 seconds.

## A-11. DETERMINATION OF SULPHATE

**A-11.0 General** — Sulphate is determined by gravimetric method using barium chloride.

**A-11.1 Reagents**

**A-11.1.1** *Barium Chloride Solution* — Dissolve 12 g of barium chloride in sufficient water to make 100 ml.

**A-11.1.2** *Dilute Hydrochloric Acid* — approximately 4 N.

**A-11.2 Procedure** — Weigh accurately about 2 g of the material and dissolve in 125 ml of water ( adding just sufficient dilute hydrochloric acid in the case of nickel carbonate ) heat the solution to boiling. Filter in a 300-ml beaker and wash until free from chloride. Add 20 ml of barium chloride solution slowly with stirring and allow to stand over night. Filter any precipitate formed, through sintered glass crucible ( G No. 4 ), wash with water until free from chloride and dry at 105°C to a constant mass.

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*Specification for sulphuric acid ( *second revision* ).

**A-11.2.1** *For Nickel Sulphamate* — To 10·0 ml of the sample solution add 300 ml of water and 30 ml of barium chloride solution, slowly with stirring. Allow to stand overnight and proceed as in **A-11.2.**

**A-11.3 Calculation**

a) Sulphate ( as $SO_4$ ), percent by mass $= \frac{41{\cdot}15\ M_1}{M}$

b) Sulphate ( as $SO_4$ ), g/l $= 41{\cdot}15\ M_1$

where

$M_1$ = mass in g of barium sulphate precipitate, and
$M$ = mass in g of the material taken for the test.

# APPENDIX B

( *Clause* 4.1 )

## SAMPLING OF NICKEL SALTS FOR ELECTROPLATING

### B-1. GENERAL REQUIREMENTS OF SAMPLING

**B-1.0** In drawing, preparing, storing, and handling samples, the following precautions and directions shall be observed.

**B-1.1** Samples shall be taken in a protected place not exposed to damp air, dust or soot.

**B-1.2** The sampling instrument shall be clean and dry when used.

**B-1.3** Precautions shall be taken to protect the samples, and the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B 1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed, as thoroughly as possible, by suitable means.

**B-1.5** The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

**B-1.6** The sample containers shall be of such a size that they are almost completely filled by the sample.

**B-1.7** Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and details given under **3.2**.

## B-2. SCALE OF SAMPLING

**B-2.1 Lot** — All the containers in a single consignment of the nickel salts of the same type, same grade and belonging to the same batch of manufacture, shall constitute a lot.

**B-2.2** For ascertaining the conformity of the material in the lot to the requirements of the specification, samples shall be tested for each lot separately.

**B-2.3** The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with Table 3.

### TABLE 3 SCALE OF SAMPLING

| No. of Containers in the Lot $N$ | Sample Size $n$ |
|---|---|
| (1) | (2) |
| Up to 15 | 3 |
| 16 to 40 | 4 |
| 41 ,, 110 | 5 |
| 111 ,, 180 | 6 |
| 181 ,, 300 | 7 |
| 301 ,, 500 | 8 |
| 501 ,, 800 | 9 |
| 801 and above | 10 |

**B-2.3.1** The containers shall be selected at random. In order to ensure the randomness of selection, a random number table shall be used. For guidance and use of random number tables, IS : 4905-1968* may be referred. In the absence of a random number table, the following procedure may be adopted:

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*Methods for random sampling.

> 'Starting from any container in the lot, count them as 1, 2, 3... up to $r$ and so on in one order, where $r$ is an integral part of $N/n$. Every $r$th container thus counted shall be withdrawn to constitute the sample.'

## B-3. PREPARATION OF TEST SAMPLES

**B-3.1** Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected according to **B-2.3**. The total quantity of the material drawn from each container shall be sufficient to make triplicate determinations for all the characteristics given in the specification.

**B-3.2** Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal quantity of material shall be taken from each selected container and shall be well mixed up together so as to form a composite sample. The quantity of material for the composite sample shall be sufficient to make triplicate determinations for all the characteristics tested on the composite sample ( *see* **B-4.2** ). The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

**B-3.3** The remaining portions of the material from each container ( after the quantity needed for the composite sample has been taken ), shall be divided into three equal parts. These parts shall be immediately transferred to separate bottles and labelled with all the particulars of sampling given in **B-1.7**. The material in each such bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in the such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

**B-3.4 Referee Sample** — The referee sample shall consist of the composite samples ( *see* **B-3.2** ) and a set of individual samples ( *see* **B-3.3** ) marked for this purpose and shall bear the seals of the purchaser and supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

## B-4. NUMBER OF TESTS

**B-4.** Tests for the determination of nickel, cobalt, copper, iron, and zinc contents of the nickel salts shall be carried out on each of the individual samples.

**B-4.2** Tests for determination of all other characteristics of the nickel salts shall be carried out on the composite sample.

## B-5. CRITERIA FOR CONFORMITY

**B-5.1 Individual Samples** — For those characteristics which are tested on individual samples, the mean and range of test results shall be computed as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{sum of individual test results}}{\text{Number of tests}}$$

$\text{Mean } (\bar{R})$ = difference between the maximum and minimum values of test results.

**B-5.1.1** The lot shall be declared to have satisfied the requirements for the following characteristics of nickel salts and nickel sulphamate solution if the corresponding criteria for conformity given against the characteristics are fulfilled:

| *Characteristics* | *Criteria for Conformity* |
|---|---|
| Nickel | $\bar{X} - 0{\cdot}6\ R$ is greater than or equal to the corresponding minimum value specified. |
| Cobalt, Copper, Iron and Zinc | $\bar{X} + 0{\cdot}6\ R$ is less than or equal to the corresponding maximum value specified. |

**B-5.2 Composite Sample** — For declaring the conformity of the lot to the requirements of all other characteristics of the nickel salts and nickel sulphamate solution, all the test results on the composite sample shall meet the corresponding requirements specified.

**B-5.3** The lot shall be declared as conforming to the requirements of the specification if **B-5.1.1** and **B-5.2** are satisfied.

## INDIAN STANDARDS ON ELECTROPLATING CHEMICALS

| IS: | |
|---|---|
| 330-1968 | Chromium trioxide (*first revision* ) |
| 1809-1979 | Nickel salts for electroplating ( *second revision* ) |
| 1880-1977 | Zinc oxide and zinc salts for electroplating ( *first revision*) |
| 2214-1977 | Silver nitrate, pure and analytical reagent (*first revision* ) |
| 3026-1968 | Tin salts for electroplating (*first revision* ) |
| 4846-1968 | Sodium potassium tartrate ( Rochelle salt ) |
| 4847-1978 | Copper salts for electroplating (*first revision* ) |
| 5003-1968 | Cadmium oxide for electroplating |
| 5761-1970 | Gold cyanide and gold potassium cyanide for electroplating |
| 6267-1971 | Silver cyanide and silver potassium cyanide for electroplating |
| 6358-1971 | Potassium and sodium cyanides for electroplating |
| 6588-1972 | Gold chloride ( chlorauric acid ) |
| 7038-1973 | Chloroplatinic acid ( platinic chloride ) |
| 7067-1973 | Fluoboric acid and metal fluoborates for electroplating |
| 8590-1977 | Indium sulphate for electroplating |
| 9341-1979 | Sulphamic acid for electroplating |
| 9342-1979 | Sodium hypophosphite for electroless plating |

# INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

**Base units**

| *Quantity* | *Unit* | *Symbol* |
|---|---|---|
| Length | metre | m |
| Mass | kilogram | kg |
| Time | second | s |
| Electric current | ampere | A |
| Thermodynamic temperature | kelvin | K |
| Luminous intensity | candela | cd |
| Amount of substance | mole | mol |

**Supplementary Units**

| *Quantity* | *Unit* | *Symbol* |
|---|---|---|
| Plane angle | radian | rad |
| Solid angle | steradian | sr |

**Derived Units**

| *Quantity* | *Unit* | *Symbol* | *Definition* |
|---|---|---|---|
| Force | newton | N | 1 N = 1 kg. $m/s^2$ |
| Energy | joule | J | 1 J = 1 N.m |
| Power | watt | W | 1 W = 1 J/s |
| Flux | weber | Wb | 1 Wb = 1 V.s |
| Flux density | tesla | T | 1 T = 1 $Wb/m^2$ |
| Frequency | hertz | Hz | 1 Hz = 1 c/s ($s^{-1}$) |
| Electric conductance | siemens | S | 1 S = 1 A/V |
| Electromotive force | volt | V | 1 V = 1 W/A |
| Pressure, stress | pascal | Pa | 1 Pa = 1 $N/m^2$ |

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